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SIMPLE ION-EXCHANGE SYSTEM FOR TRANSPLUTONIUM  
ELEMENTS SYNTHESIZED BY HEAVY-ION BOMBARDMENTS

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SIMPLE ION-EXCHANGE SYSTEM FOR TRANSPLUTONIUM ELEMENTS  
SYNTHESIZED BY HEAVY-ION BOMBARDMENTS

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A simple system has been developed for rapid ion exchange separation followed by source preparation for alpha counting, which is applicable for various kinds of solvent (such as strong acids and organic solvents) at a given temperature (up to 120 °C) and pressure (up to 4 MPa).

By use of the system,  $^{250}\text{Fm}$  ( $T_{1/2}$  : 30 min) and  $^{246}\text{Cf}$  ( $T_{1/2}$  : 35.7 hr) synthesized by the  $^{16}\text{O} + ^{238}\text{U}$  reaction were separated quantitatively from a uranium target and an aluminium catcher foil.

This report deals with the outline of the system and its application, and contains as appendixes manuals for the system preparation and handling.

Keywords: Pressurized Ion-exchange, Rapid separation, Transplutonium,  
Simple system, Fluoroplastic tube,  $\alpha$ -Source Preparation,  
 $^{250}\text{Fm}$ ,  $^{246}\text{Cf}$ ,  $^{16}\text{O} + ^{238}\text{U}$ .

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重イオン照射により合成される超プルトニウム元素のための  
簡単なイオン分離交換システム

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(1986年12月15日受理)

重イオン照射により合成される超プルトニウム元素の迅速分離及び $\alpha$ 放射能測定用試料の調製を行うための簡単なイオン交換分離システムを開発した。本システムは、一定の温度(120℃以下)圧力(4MPa以下)のもとで強酸、有機溶媒等種々の溶液を溶離液として用いることができる。

本システムを用いて、塩酸系陽イオン交換法により $^{16}\text{O} + ^{238}\text{U}$ 反応で合成された $^{250}\text{Fm}$ ( $T_{1/2}$ :30min)及び $^{246}\text{Cf}$ ( $T_{1/2}$ :37.5hr)をウラン・ターゲット及びアルミニウム・キャッチャーフォイルから迅速に分離定量した。

本報告書は、システムの概要及びその応用例について述べたものであり、付録としてセットアップ及び操作法についての詳細なマニュアルを載せた。

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## 1. Introduction

Many transplutonium nuclides synthesized by heavy-ion bombardments have relatively short half-lives and emit alpha particles. The production yields of the nuclides are quite low mainly owing to their small formation cross sections. Accordingly, rapid separation of the transplutonium elements and immediate preparation of the sources for alpha-ray spectrometry are necessarily required with considerable recovery in order to investigate the nuclear reaction.

Among various rapid chemical separation systems<sup>1-7</sup>, the continuous solvent extractor, SISAK<sup>6,7</sup>, must be the most excellent system for gamma- and beta-radioactive nuclides, but it might associate with a serious difficulty in source preparation for the alpha-radioactive nuclides. Ion exchange method is suitable for rapid separation, concentration and alpha-counting source preparation of such transplutonium nuclides. If many kinds of solvents are utilized as eluents, wide separation systems by ion exchange will become available.

A pressurized ion exchange apparatus has been devised, which is applicable for various solvents at a given temperature and flow rate. By evaporation of the effluents directly on a tantalum disk, the alpha-counting sources were immediately prepared. The system has been applied to cation exchange separation for the transplutonium nuclides produced by the  $^{16}\text{O} + ^{238}\text{U}$  reactions.



## 2. Experimental

### 2.1 Reagents

The strongly acidic cation exchange resin, MCI GEL CK08Y ( particle size:  $23.5 \pm 4 \mu\text{m}$ , crosslinking: 8 %, exchange capacity:  $\geq 1.9 \text{ meq/ml resin}$  ), was obtained commercially by Mitsubishi Chemical Industries Limited. The resin was washed successively with methyl alcohol, hydrochloric acid and distilled water before use.

Water and reagent-grade hydrochloric acid used as eluents were purified by distillation in quartz glass apparatus in order to remove any salt-deposited material.

### 2.2 Radioactive tracers

Tracers of  $^{241}\text{Am}$ ,  $^{244}\text{Cm}$  and long-lived fission products, which were separated from spent fuel solution by anion exchange with hydrochloric acid<sup>8</sup>, were used to obtain the elution curves.

$^{250}\text{Fm}$  and  $^{246}\text{Cf}$  were synthesized by bombardments of  $^{16}\text{O}$  ion beams with energy 88 MeV to  $^{238}\text{U}$  target ( ca. 1.4 mg ) using the JEARI Tandem Accelerator<sup>9,10</sup>. The beam current was about 150 particle nA and the irradiation duration was 30 minutes. The recoil nuclides contained  $^{250}\text{Fm}$  and  $^{246}\text{Cf}$  were caught on aluminium backing foil ( ca. 4 mg ).

### 2.3 Activity measurements

The alpha and beta activities were measured with silicon surface barrier detector. The counting sources prepared on

tantalum disks were positioned at a distance of about 5 mm from the detector to gain higher detection efficiency ( 10-20 % ). A Ge(Li) detector was also used to identify gamma-radioactive nuclides.

### **3. Ion-exchange separation system**

#### **3.1 Column for the system**

The ion-exchange column used for the system ( see Fig.1 and Photo.1 ) was prepared by packing the resin into fluoroplastic tube ( Diflon or Teflon, inner diameter: 1.5 or 2 mm, column volume: 0.25 ml ) and compacting with pressurized nitrogen gas ( ca. 1 MPa ). The resin was fixed with quartz wool plugs at the both ends.

#### **3.2 Component of the system**

The ion-exchange separation system was composed of the following parts ( see Fig. 2 ):

- 1) controller for flow rate of eluents ( a nitrogen gas tank, a pressure control valve and a 3-way valve made of Teflon ),
- 2) reservoir for sample solution and eluents ( a coupled 4-way valve made of Teflon, two spiral Teflon tubes ( i.d.: 2 mm, volume: ca.10 ml ) and a tube pump for sucking the solution )( see Photo.2 ),
- 3) ion exchange separator ( a column and a thermostat such as a water bath and an air bath ) ( see Photo.3 ),
- 4) monitor, if necessary, for gamma or beta activities ( a well-type NaI(Tl) scintillation detector or a NA102A plastic

tantalum disks were positioned at a distance of about 5 mm from the detector to gain higher detection efficiency ( 10-20 % ). A Ge(Li) detector was also used to identify gamma-radioactive nuclides.

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- 4) monitor, if necessary, for gamma or beta activities ( a well-type NaI(Tl) scintillation detector or a NA102A plastic

scintillation tube detector ) ( see Photo.4 ), and

- 5) evaporator for effluent ( a hot plate with quartz glass and, if necessary, a fraction collector with a drop counter ) ( see Photo.5 ).

All parts were connected through Teflon tubes ( i.d.: 0.25 or 0.5 mm ).

### 3.3 Operation

After dissolution of a bombarded target and a catcher foil with an appropriate acid solution, the separation of elements in question is carried out as follows.

- 1) A definite volume of the first eluent is previously sucked into the spiral tube-I by the tube pump, and then the sample solution is consecutively sucked into the same spiral tube. Small volume of air is also intentionally sucked between two solutions to avoid mixing of those.
- 2) The coupled 4-way valve is turned in order to transfer the solutions in the spiral tube-I to the column. The sample solution is then fed to the column followed by the first eluent with nitrogen gas pressure by manipulating the 3-way valve. During this step, the second eluent is sucked into the spiral tube-II.
- 3) The same operation is repeated interchangeably with the two spiral tubes for successive eluents.
- 4) The eluted drops are directly deposited at the centre of the heated tantalum disk ( 24 mm $\phi$  x 0.05 mm ), being evaporated

moderately by careful control of the hot plate temperature.

The flow rate can be adjusted by controlling the nitrogen gas pressure ( 0.1-4 MPa ) to the system. The column is thermostated at  $90 \pm 1^{\circ}\text{C}$ .

### 3.4 Characteristics of the system

The solution in the system contacts with only fluoroplastic material ( Teflon and Diflon ) and the ion-exchange resin. The system is therefore applicable for various kinds of solvent unless they react with the fluoroplastic material, and endurable with elevated temperature up to  $120^{\circ}\text{C}$  ( decomposition of the resin ) and high pressure up to about 4 MPa ( bursting the Teflon and Diflon tubes ). Consequently, it is possible to carry out rapid ion-exchange separation with many kinds of eluents such as hydrochloric, nitric and fluoric acids, organic solvents and the mixture of those under the condition of the pressure below 4 MPa and the temperature below  $120^{\circ}\text{C}$ .

The flow of eluent is pulseless. The small amounts of eluent ( more than 0.1 ml ) is passed through the column without mixing with another eluent and can be deposited on a tantalum disk.

The system is simply operated by handling the two valves and the tube pump.

## 4. Application

### 4.1 Elution scheme

Cation exchange separation of transplutonium elements with hydrochloric acid solutions was examined using the system. Figure 3 shows elution curves for separation of Am and Cm tracers from fission products, 1.1 mg of U ( target material ) and 3.2 mg of Al ( catcher foil ) at 90°C and flow rate of 22-30 cm/min. The excess Al over the resin capacity was passed through the column with 1M HCl solution, and then Cs, U, Sr and the residual amounts of Al were eluted. The transplutonium elements were eluted with 4M HCl accompanying with the rare earths. To avoid the aluminium contamination in transplutonium fraction, initial several drops of 4M HCl effluent should be discarded.

The recovery of the transplutonium elements was more than 90 %. It took about 15 minutes to separate the transplutonium elements and prepare the alpha-counting source.

### 4.2 Separation of $^{250}\text{Fm}$ and $^{246}\text{Cf}$

Based on the result mentioned above, rapid separation of the transplutonium nuclides synthesized by the  $^{16}\text{O} + ^{238}\text{U}$  reaction<sup>9,10</sup> was carried out.

A bombarded uranium target ( ca. 1.4 mg ) with an aluminium backing ( ca. 4 mg ) was dissolved with concentrated HCl in 5-ml beaker under an infrared lamp. Concentrated  $\text{HNO}_3$  was added to the beaker in order to completely dissolve the sample and oxidize U (IV) and the other actinide elements. The solution was heated to

near-dryness. The residue was redissolved in small volume of  $\leq 1$ M HCl solution. Separation of the transplutonium nuclides was performed according to the flow diagram shown in Fig. 4.

The counting source on the tantalum disk was prepared by igniting it in gas burner flame and alpha-ray spectrum of the source immediately was measured. Figure 5 shows alpha-ray spectrum of the transplutonium fraction.  $^{250}\text{Fm}$  ( $T_{1/2}$ : 30 min, alpha energy: 7.43 MeV)<sup>11</sup> and  $^{246}\text{Cf}$  ( $T_{1/2}$ : 35.7 hr, alpha energy: 6.76 MeV)<sup>11</sup> were identified by their alpha particle energies and their half-lives<sup>9</sup>.

## 5. Conclusions

A system has been developed for ion exchange separation followed by immediate source preparation for alpha counting. The manipulation of the system is very simple. The system was applied to rapid separation of the transplutonium elements synthesized by heavy-ion reaction from an aluminium catcher foil and a uranium target. The time for the separation the counting source preparation was about 15 minutes.

The system can be used with various kinds of solvent ( such as strong acids and organic solvents ) at elevated temperature ( up to  $120^{\circ}\text{C}$  ) and pressure ( up to 4 MPa ), so that wide application would be expected for rapid separation of short-lived elements using ion exchange with mixed media of mineral acids and organic solvents<sup>10,12,13</sup>.

We have been devised an automatic ion-exchange separation apparatus designed on the base of this system, which attaches to

near-dryness. The residue was redissolved in small volume of  $\leq 1$ M HCl solution. Separation of the transplutonium nuclides was performed according to the flow diagram shown in Fig. 4.

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We have been devised an automatic ion-exchange separation apparatus designed on the base of this system, which attaches to



the R-2 beam line of the JAERI Tandem Accelerator<sup>13</sup>. The apparatus can be employed in combination with a gas-jet recoil transport system and an activity measurement system. It is promising to more rapidly separate the transplutonium nuclides synthesized by heavy-ion bombardments.

### Acknowledgments

The authors wish to thank the nuclear chemistry group of JAERI for bombardments of the  $^{16}\text{O}$  ion beams to  $^{238}\text{U}$  target by JAERI Tandem Accelerator. Thanks are also due to Drs. H. Okashita and T. Yamamoto for helpful discussion.

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## Appendix

### I. Manual for the column preparation

[ Refer to Fig.1. ]

- 1) Cut Diflon ( or Teflon ) tube ( o.d.: 3 mm, i.d.: 1.5 mm, l: ca. 16 cm ) to obtain 0.25 ml of column volumn.
- 2) Widen the both ends of the tube in trumpet shape with a solder copper after setting two nuts of joints to the tube.
- 3) Enter some plugs of quartz wool step by step ( length: ca. 1-2 cm ) into an end of the tube with a glass bar ( <1.5 mm $\phi$  ), and connect a Diflon joint ( i.d.: 0.5 mm ) with the nut.
- 4) Stuff the required volume of the resin together with water into the tube from a resin reservoir ( Photo.6 ) by nitrogen gas pressure ( ca. 1 MPa ).
- 5) Enter some plugs of quartz wool step by step ( length: ca. 1-2 cm ) into the other end of the tube, and connect a Diflon joint ( i.d.: 0.5 mm ) with the other nut.
- 6) Wash the resin with 10 ml of 4M hydrochloric acid, 5 ml of distilled water and 10 ml of methyl alcohol.
- 7) Determine the weight of the resin, if necessary, after drying at 110°C and keeping in a desiccator at room temperature for several hours.

## II. Setup of the system

[ Refer to Fig.2. ]

- 1) Fix a 3-way valve and a coupled 4-way valve made of Teflon to a burette support, and set two spiral Teflon tubes ( o.d.: 3 mm, i.d.: 2 mm, l: ca. 3.2 m ) to the coupled 4-way valve ( see Photo.2 ).
- 2) Join a tube pump and the coupled 4-way valve with a Teflon tube ( o.d.: 2 mm, i.d.: 1 mm ).
- 3) Connect a Teflon tube ( 1 mm $\phi$  x ca.40 cm ) with the coupled 4-way valve for introducing the sample solution and/or some eluents.
- 4) Set a pressure control valve ( <15 MPa ) to a nitrogen gas tank, and join this valve and the 3-way valve with a Teflon tube ( o.d.:3 mm, i.d.: 2 mm ).
- 5) Join the prepared column and the coupled 4-way valve with a Teflon tube ( o.d.:2 mm, i.d.: 0.5 mm ), and give the column a water bath or an air bath maintained at 90°C ( see Photo.3 ).
- 6) Set up, if necessary, a well-type NaI(Tl) scintillator or a NA102A scintillation tube detector ( see Photo.4 ), and join the column and the detector with a Teflon tube ( o.d.: 2 mm, i.d.: 0.5 mm ).
- 7) Join the detector and a drop counter with a Teflon tube ( o.d.: 2 mm, i.d.: 0.5 mm ).
- 8) Put a 5-ml beaker or a tantalum disk ( 24 mm $\phi$  x 0.05 mm ) on a quartz glass plate ( 10 cm x 10 cm ) heated by hot

plate, whose temperature are controlled carefully.

- 9) Open the valve of nitrogen gas tank followed by the pressure control valve. ( The gas-tank valve should shut for safty. )

### III. Separation procedure

[ Refer Fig.2, 3 and 4. ]

- 1) Dissolve a bombarded uranium target ( up to 5 mg ) with an aluminium foil ( up to 5 mg ) with 0.1-0.3 ml of concentrated HCl in a 5-ml beaker.
- 2) Heat the beaker under an infrared lamp.
- 3) Add 0.1 ml of concentrated  $\text{HNO}_3$  to the beaker, and evaporate the solution to near-dryness: the sample is completely dissolved, and U(IV) and the other actinides are oxidized.
- 4) Redissolve the residue in about 0.5 ml of  $\leq 1\text{M}$  HCl solution.
- 5) Suck previously a definite volume of the first eluent ( 2 ml of 1M HCl ) into the spiral tube-I by the tube pump. To avoid mixing of the solution with the sample solution, suck intentionally small volume of air.
- 6) Suck the sample solution into the same spiral tube.
- 7) Turn up the coupled 4-way and then turn on the 3-way valve: the sample solution and the first eluent are transferred into the column. Receive the solutions passed through the column into a 5-ml beaker. Controll the nitrogen pressure ( 0.1-4 MPa ) to keep a given flow rate.
- 8) Suck the second eluent ( 8 ml of 1MHCl ) into the spiral tube-II during flow of the solutions in the tube-I.

- 9) Turn off the 3-way valve after flowing out the solutions in the spiral tube-I.
- 10) Turn down the coupled 4-way valve and then turn on the 3-way valve to supply the second eluent into the column. Receive the effluent into another 5-ml beaker: U, Al and Sr are eluted.
- 11) Suck the third eluent ( 1 ml of 4M HCl ) into the spiral tube-I during flow of the solution in the tube-II.
- 12) Turn off the 3-way valve after flowing out the second eluent.
- 13) Turn up the coupled 4-way valve and then turn on the 3-way valve: the third eluent is transferred into the column, and the transplutonium elements are eluted accompanying with the rare earths.
- 14) Discard the initial several drops of the effluent to avoid the aluminium contamination in the transplutonium fraction. Put a tantalum disk under the drop counter, and receive the eluted drops directly at the centre of the disk.
- 15) Evaporate carefully the drops on the disk by control the hot-plate temperature.
- 16) Prepare a source for alpha-activity measurements by igniting the tantalum disk in gas burner flame for a few seconds.
- 17) Measure alpha activity of the prepared source.



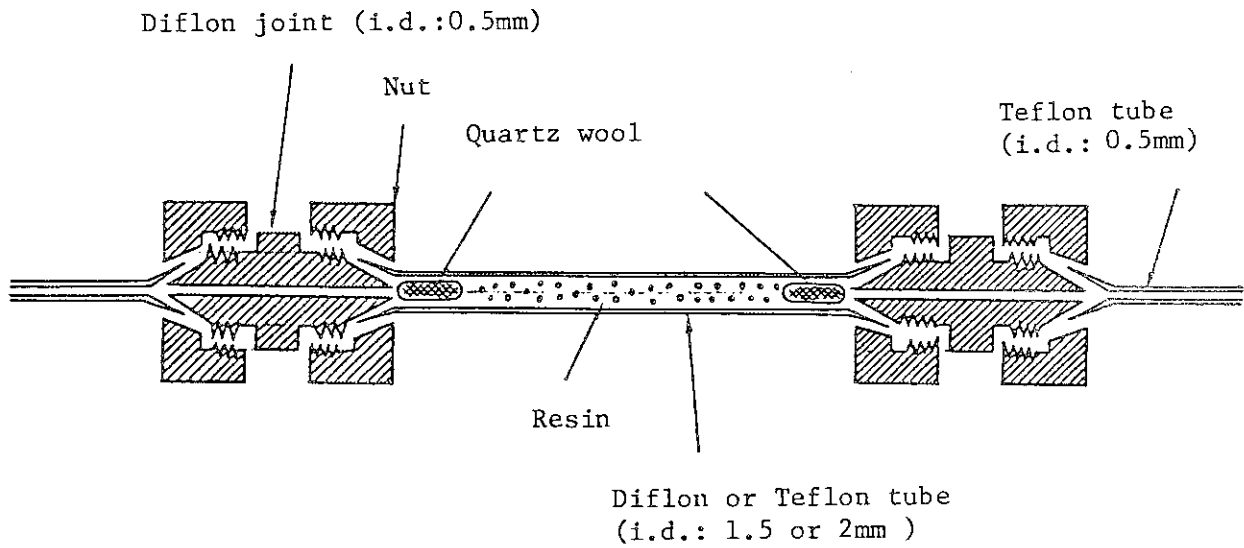


Fig.1 Diagram of the ion-exchange column.

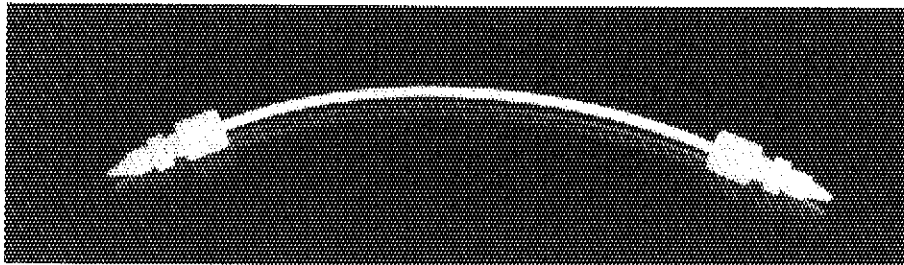


Photo.1 Prepared ion-exchange column.

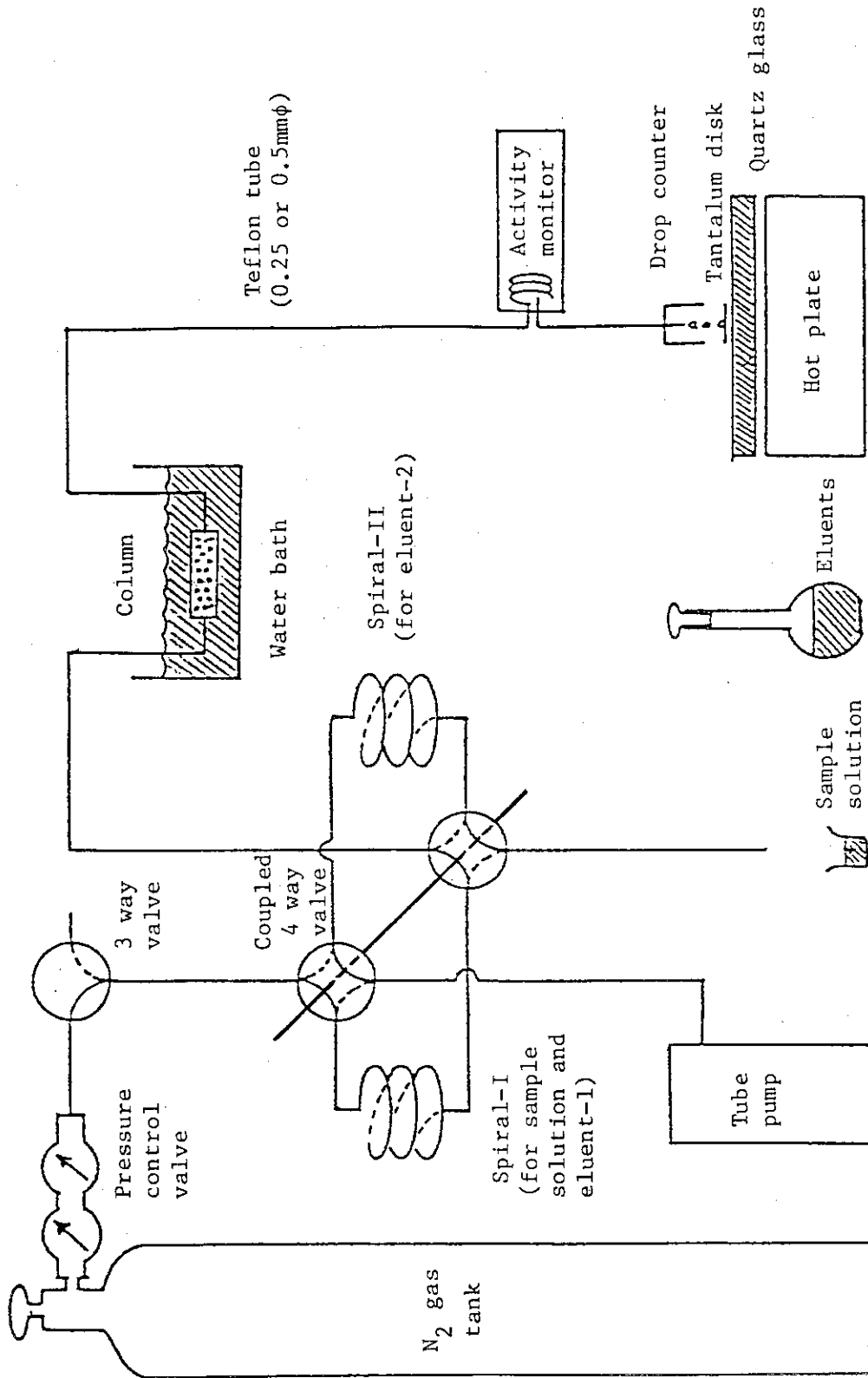


Fig. 2. Arrangement of the system.

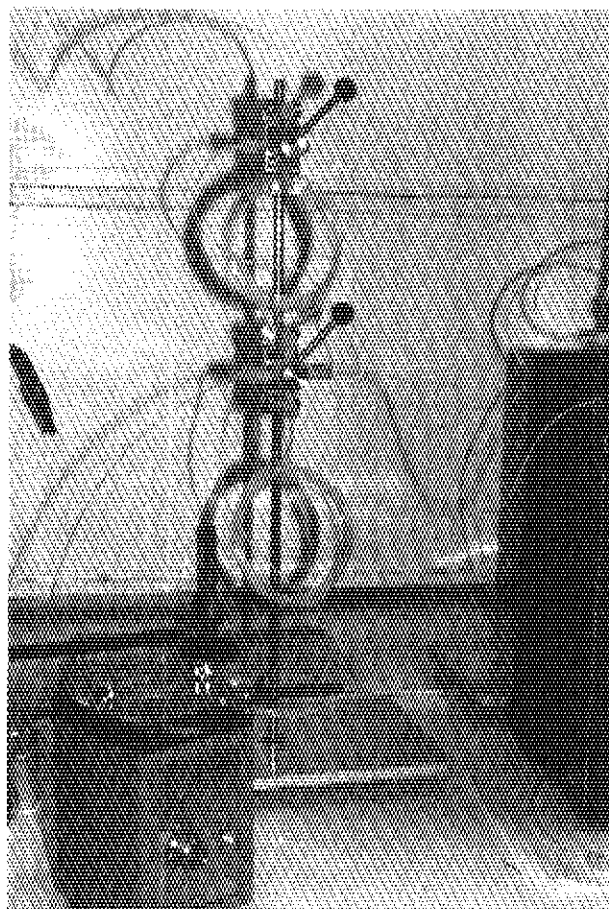


Photo.2 Reservoir for sample solution and eluents.

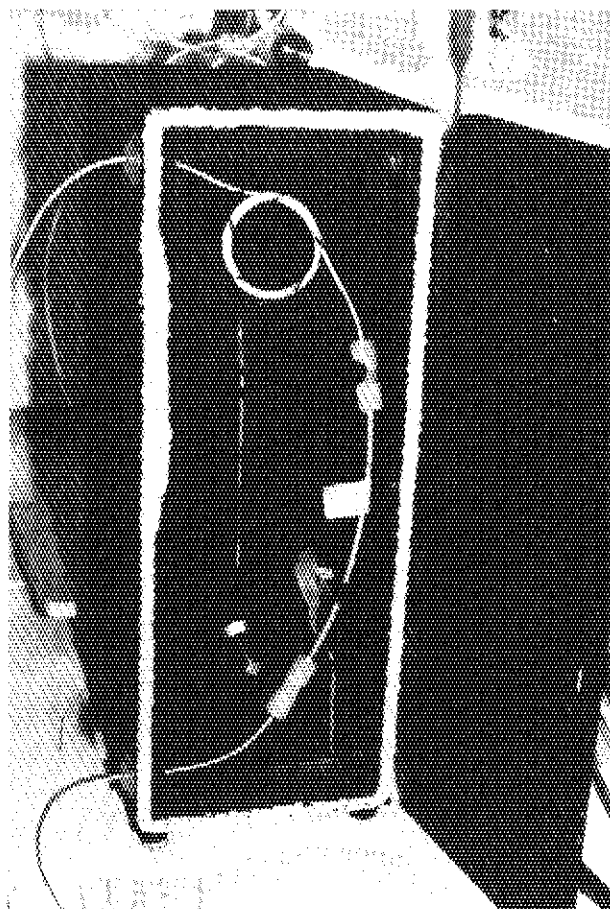


Photo.3 Ion-exchange column in a thermostat.

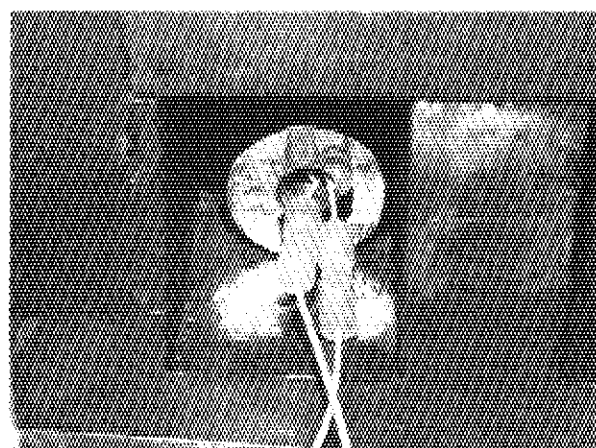
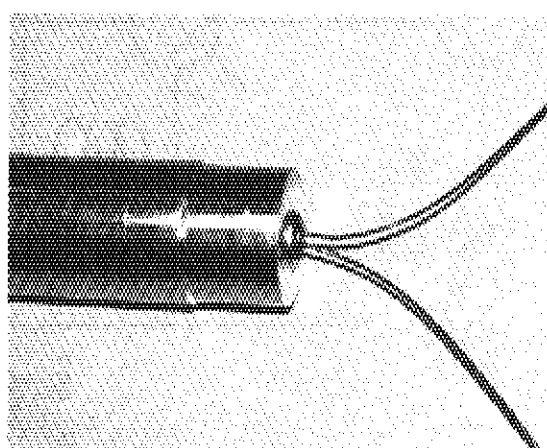


Photo.4 Monitor for gamma or beta radioactivities: a well-type NaI detector (right) and a NA102A tube detector (left).



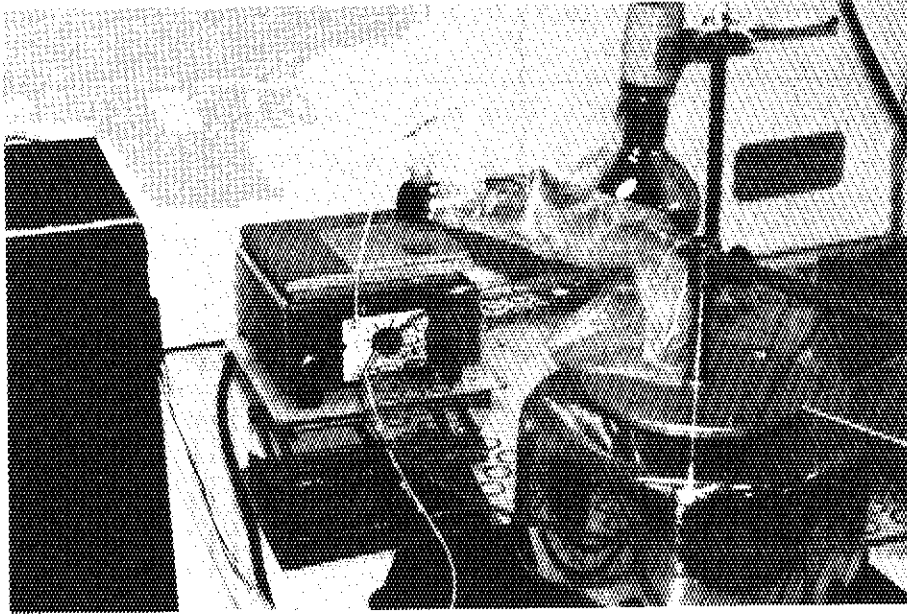


Photo.5 Evaporator for effluent and a fraction collector.

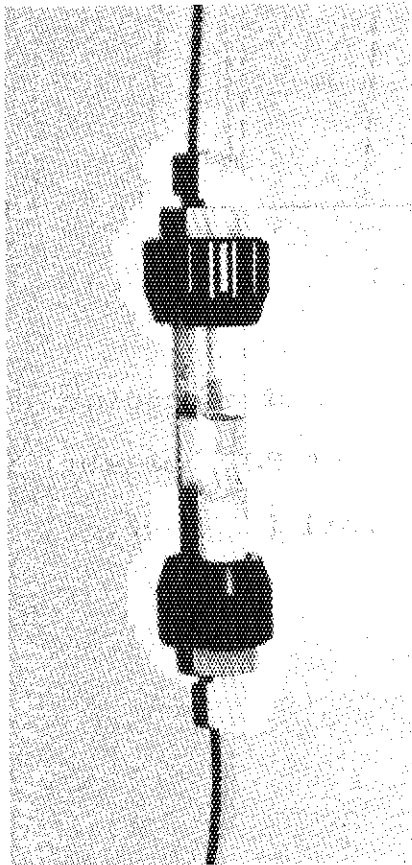


Photo.6 Resin reservoir for  
column preparation.

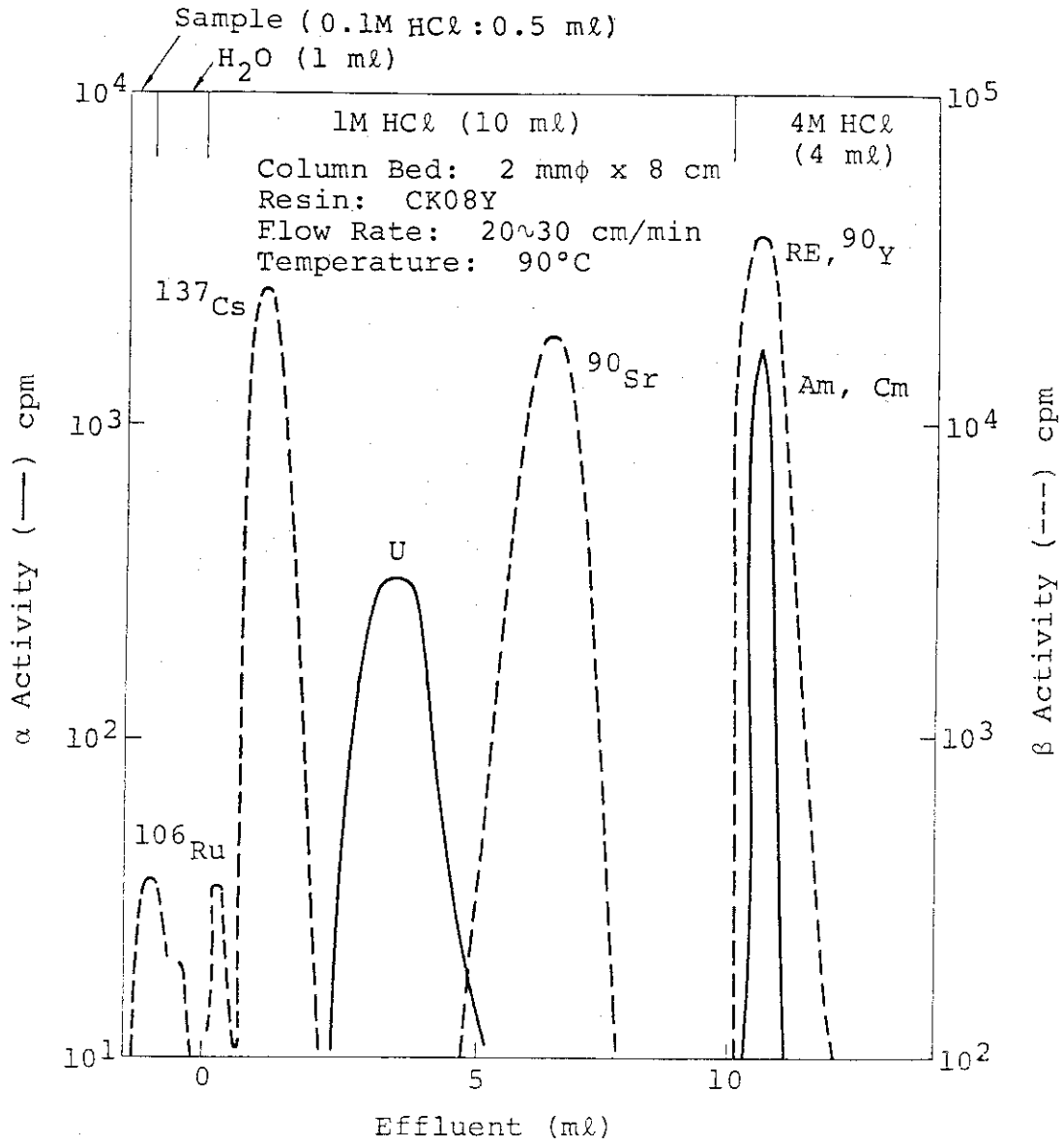


Fig. 3. Elution curves for separation of transplutonium elements from the mixture of F.P., uranium target ( 1.1 mg ) and aluminium foil ( 3.2 mg ).

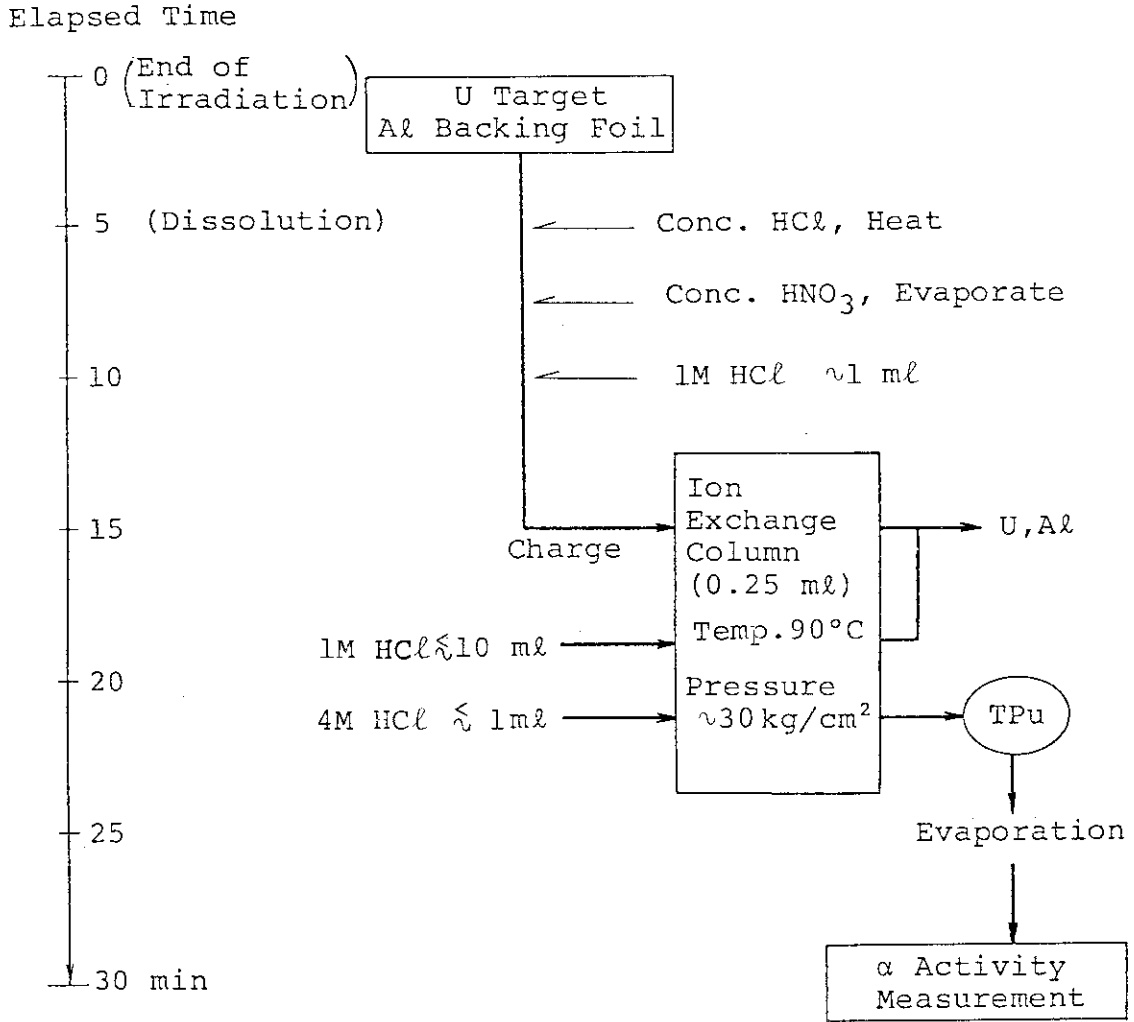


Fig. 4. Flow diagram for the rapid ion exchange separation of transplutonium elements from heavy-ion bombarded uranium target with aluminium foil.

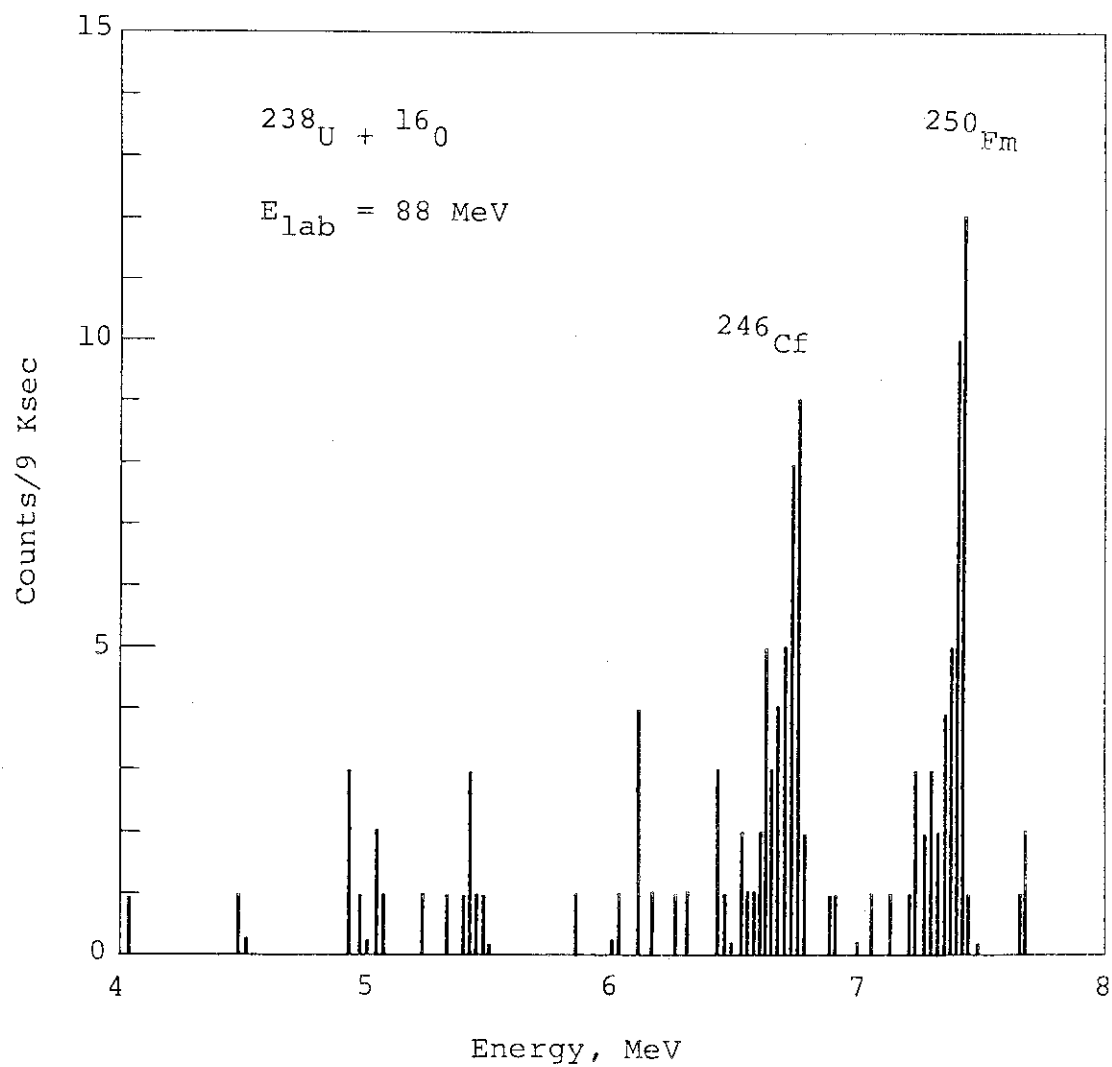


Fig. 5. Alpha-ray spectrum of the transplutonium fraction.